GREAT deal has been published during the past three years regarding the various sources of natural rubber which have been called upon to supplement this nation's stockpile and to meet those special needs for which commercial synthetic rubbers are not entirely satisfactory. We have read of the wild rubbers being brought in under great difficulty from Central and South America, of the 32,000 acres of guayule grown by the government, largely in California, and now nearly ready for harvest, and of the large experimental plantings of Cryptostegia that were made in Haiti. Almost nothing, however, has appeared regarding a plant which is known to be potentially rich in high-grade rubber and which has been extensively cultivated outside this country. This is the Russian dandelion, or Taraxacum kok-saghyz.

In the course of a systematic investigation instituted in Russia in 1929 to determine the possibilities of producing natural rubber in that country, the kok-saghyz was found near Tien Shan, Kazakstan, near the border of China. By 1932 more than 2,000 acres of this plant were under cultivation in Russia, and it is reported that at the time of the German invasion of Russia approximately 200,000 acres of kok-saghyz had been planted for the production of rubber.

Shortly after the tragedy at Pearl Harbor the United States Government started negotiations with Russia to obtain kok-saghyz seed. These eventuated in the arrival in Washington on May 8, 1942, of two gunny sacks of seed flown from Kuibyshev. On the same day the seed arrived some of it was repackaged and distributed by air to various points in the United States, where more than 60 indicator plantings were established through cooperation with state agricultural experiment stations.

It was shortly prior to this time that the Emergency Rubber Project was authorized by Congress. It was "An Act to provide for the planting of guayule and other rubberbearing plants and to make available a source of crude rubber for emergency and defense uses . . ." The responsibility for the Emergency Rubber Project was delegated by the Secretary of Agriculture to the Forest Service. The Forest Service in turn called upon other bureaus in the Department for assistance in certain phases of the work. The task of carrying out agronomic and plant improvement research was assigned to the Bureau of Plant Industry, Soils, and Agricultural Engineering. The Bureau of Agricultural and Industrial Chemistry was charged with the responsibility for developing methods for recovering the rubber from these plants, for determining the technical feasibility of such methods by pilot-plant operations, for evaluating the rubber obtained, and for determining the economic factors involved in rubber recovery. These responsibilities were in turn delegated to the Eastern Regional Research Laboratory at Philadelphia. Thus, when the first kok-saghyz roots were harvested in the Fall of 1942, they were shipped to the Laboratory at Philadelphia for rubber-recovery studies.

In spite of the late planting of the seed and the unsatisfactory nature of some of the planting sites, sufficient roots were obtained to permit working out on a batch pilot-plant scale a comparatively simple process for recovering the rubber. A large-scale continuous pilot-plant unit was designed and erected at the Philadelphia Laboratory, and during the Winter of 1943 and 1944 a sufficient quantity of the rubber was produced to permit its commercial evaluation. The roots for these large-scale experimental operations were derived largely from more than 600 acres of kok-saghyz planted by the Forest Service, principally in Michigan and Minnesota.

Analysis of typical one-season kok-sughyz plants showed

Roderick K. Eskew²

that more than 90% of the rubber is in the form of latex in interconnecting ducts in the root itself. The remainder is in the crown and leaves of the plant, which constitute about 25% of its dry weight. This leaf and crown fraction, however, is rich in pectin, containing nearly 40% of the total pectin in the plant. Since pectin interferes with rubber recovery, the tops were mechanically cut off in the field just before harvesting. This practice also reduces the bulk to be handled in the factory.

The rubber-recovery process described here thus applies only to the roots of kok-saghyz. Individual kok-saghyz roots having as much as 23% rubber hydrocarbon on the dry basis have been grown in this country. Such a high rubber content, however, is exceptional. Most of the roots processed in the pilot plant contained between 5% and 10% rubber hydrocarbon on a dry basis. The roots are also rich in carbohydrates; the amount depends, among other things, on the time of harvest; it ranges from 10% in roots harvested in the spring to 45% in fall-harvested roots.

Before discussing the recovery process for kok-saghyz rubber, it is necessary to give some consideration to the localities in which the roots may be grown and the seasons of harvesting. Although much remains to be done toward perfecting methods for growing kok-saghyz of high rubber content and high yields per acre, it has been grown experimentally during the winter months in certain parts of Florida and during the summer in the Red River Valley of Minnesota, in certain sections of Michigan, Wisconsin, and Montana.

For reasons of simplicity the following discussion will be based upon probable growing conditions in Minnesota or Michigan. It is assumed that the seed would be planted in the field as early as possible in the spring, preferably prior to June first. Harvesting of approximately 50% of the roots planted would take place between October 15 and November 15. The remaining roots would be left in the ground until the following spring and would be harvested as nearly as possible between April 15 and May 15, with the next seeding following immediately.

Roots left in the ground during the winter generally increase percentage-wise in rubber, even when harvested early the following spring. This condition is undoubtedly due in part to a corresponding loss of carbohydrates resulting from renewed top growth.

In order to preserve the carbohydrates during storage of harvested roots, the full six months' supply harvested in the fall, and a similar quantity harvested in the spring, would have to be dried during each harvesting period. The roots could be washed by simple devices already worked out for field operations. They could then be spread on the ground to a depth of approximately one foot and left to dry, with occasional turning if necessary, to as low a moisture content as weather conditions permit. This practice is necessary to minimize the drying capacity that would be required in a factory drying roots immediately after washing; such roots contain about 85% moisture.

After drying, some roots would go immediately into process, and the remainder would be baled for storage.

Rubber-Recovery Process

The first step of the process consists in countercurrently leaching the dried roots in hot water to remove the carbohydrates. This produces a solution which can be fermented directly to alcohol. A small amount of soda ash is used during leaching to soften the roots. The leached roots, now only about 55% of their original dried weight, are given a short pebble milling in water to disperse the softened plant tissue

 [&]quot;Natural Rubber from Domestic Sources." Paper No. 11.
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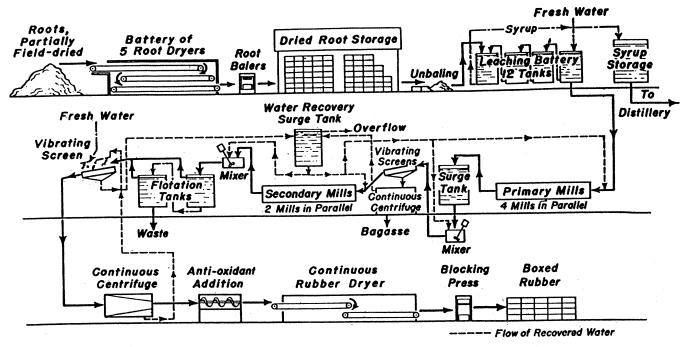


Fig. 1. Flow Diagram for Kok-saghyz Factory—10 Long Tons of Rubber Per Day

and to roll together the fine filaments of rubber. By diluting this slurry and screening it, 65% of the remaining solids are eliminated, leaving largely rubber and adhering root skins. A second short pebble milling with water disengages the root skins from the rubber, and the two are separated by a subsequent flotation step wherein the rubber floats and the plant debris (bagasse) sinks. A final washing of the rubber on a vibrating screen removes further traces of plant material. After being centrifuged to remove excess water, the rubber is mixed with an antioxidant and then dried in a through-circulation continuous drier. It is then baled and boxed in the conventional manner. A flow sheet of the process is shown in Figure 1.

Root Drying and Storage

Freshly washed roots or roots which have been partly field dried can be satisfactorily dried to the desired final moisture of approximately 15% in a continuous through-circulation belt-type drier at temperatures up to 200° F., preferably with the relative humidity maintained at approximately 10%.

The dried roots are baled and stored. They may be kept up to six months or more without any change in the quantity or quality of recoverable rubber. Some loss in carbohydrates can be anticipated, however, when storage is continued beyond three or four months.

Leaching

Leaching is carried out countercurrently in a battery of 10 to 12 tanks using water of 200-212° F. containing about 0.1% soda ash. The function of leaching is threefold:

- (1) When conducted countercurrently on fall-harvested roots, it removes the carbohydrates and recovers them in such a concentration as to provide a by-product material fermentable to alcohol or to lactic acid.
- (2) It reduces the dry weight of material going to the rubber-recovery system by eliminating soluble solids.
- (3) The soda ash used in the early stages of leaching softens the root and facilitates pebble milling.

Milling and Screening

Although rubber can be isolated from leached kok-saghyz roots by a pebble-milling process alone, it is much simpler and more economical to intersperse a dilution and a screening step between two continuous pebble-milling operations. If rubber were isolated by milling alone, the total milling time would be three or more times as great as that required for two-stage milling with intermediate dilution and screening.

In the primary milling operation the object is to roll together the coagulated filaments of rubber and to disperse plant material, particularly the softened pulpy portions of the roots, so that on dilution they can be eliminated by screening. This can ordinarily be accomplished by milling for 15 minutes in a continuous pebble mill with a ratio of 20 parts of water to one part of solids. The temperature of the mill slurry is maintained at approximately 100° F. by heating the water going into the mill. This elevated temperature softens plant tissues and reduces the consistency of the slurry, thereby facilitating milling.

The completeness of primary milling can be judged by examining the weblike rubber masses emerging from the mill. When these are stretched and examined under low-power magnification, secondary phloëm can be readily observed. Primary milling should be carried to the point where the web of rubber becomes flattened and, when stretched, reveals almost complete elimination of secondary phloëm. At this stage approximately 50% of the total rubber floats when the slurry is diluted to about 100/1.

About 65% of the root solids can be removed by screening the slurry from the primary mill after diluting it with 100 parts of water to one part of solids. A twofold purpose is accomplished by this operation. First, the subsequent milling load is reduced by 65%, and furthermore less plant material remains to contaminate the rubber. The screening operation is a very important step, and unless it eliminates at least 60% of the solids, secondary milling will be adversely affected, and a greatly increased milling time will be necessary to cause the rubber to float. The factors affecting good screening are (1) kind of roots, (2) effectiveness of leaching and proper control of pH during leaching, (3) completeness of primary milling, (4) slope, stroke, mesh, and weave of screens, and (5) rate of feed to the screens. The water passing through the screens contains approximately 0.6% of plant material. It can be clarified in a continuous centrifuge, the water reused, and the solids recovered if they prove to have by-product value.

Since most of the material passing over the screen is rubber and adhering root skins, the function of secondary pebble milling becomes one of detaching fragments of root skins in order that they may be eliminated in a subsequent flotation step. Secondary milling is ordinarily carried out for about 22 minutes at a water to solids ratio of 22:1; the temperature of the slurry is maintained at about 75° F. Higher temperatures than this tend to soften the rubber, resulting in the formation of balls and entrapment of foreign material. The yield of rubber in the recovery process de-

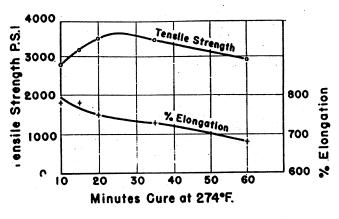


Fig. 2. Curing Curve for Kok-saghyz Rubber

pends upon how effectively it can be floated away from plant debris. Since its floatability depends upon the degree to which non-rubber constituents have been eliminated, the completeness of secondary milling is judged by how completely the rubber floats when a sample of the slurry from the mill is diluted with a large excess of water.

Flotation

The flotation step, which comes immediately after the secondary milling, permits the rubber to float away from the plant debris, which sinks. The effluent slurry from the secondary pebble mill, diluted to about 100 parts of water per one part of solids, is introduced at the center and beneath the liquid level in a continuous flotation tank. Because some of the plant materials sink very slowly, the rate of slurry feed to the flotation tanks is relatively slow. The rubber is skimmed continuously from the surface of the tank by slow-moving blades that dip about two inches below the level of the liquid. At the bottom of the same shaft is a revolving scroll, which continuously moves the debris to the discharge outlet at the center of the tank. It is pumped from this outlet to a secondary flotation tank, designed exactly like the first, where any rubber entrapped in the sediment is freed to float. Two tanks in series should recover practically all the rubber if secondary milling has been properly performed. Otherwise undermilled rubber, which does not float, is lost in the bagasse.

The floated rubber is rinsed with water sprays on a vibrating screen preparatory to drying.

Rubber Drying

Because of the physical state in which kok-saghyz rubber is recovered, it lends itself readily to rapid drying at relatively high temperatures without deterioration. Prior to drying, it is desirable for reasons of heat economy to free the rubber of as much water as possible by centrifuging. The moisture content can be reduced to between 40 and 48% without causing the rubber to stick together if the centrifugal force is kept below 100 times that of gravity.

To prevent the rubber from being degraded during drying and to enhance the physical properties of the vulcanizate, it is customary to add an antioxidant to the dewatered rubber. 4-4'-diaminodiphenylmethane is an effective agent, and it is customarily added in the form of an aqueous emulsion, which is sprayed on the rubber. A sufficient quantity is added to give a concentration of approximately 0.45% on the dry-rubber basis.

The rubber is dried in a continuous through-circulation belt-type drier at a temperature of approximately 200° F. The time required is about 45 minutes. Kok-saghyz rubber, when dried in this manner, has better physical properties than when dried slowly under vacuum. The rubber is baled and boxed in the conventional manner.

Physical and Chemical Properties of Kok-saghyz Rubber

Rubber produced by the foregoing process had the following average analysis:

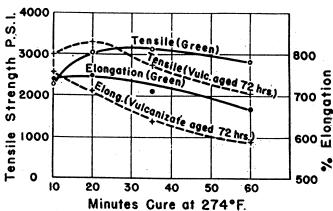


Fig. 3. Aging of Kok-saghyz Vulcanizate

	%
Rubber hydrocarbon (by bromination)	80.5
Benzol-insoluble material (by direct determination)	12.5
Acetone-soluble material (by difference)	7.0

The benzol-insoluble material can be reduced to 5% or less by an additional pebble mill scrubbing, but unlike the benzol-insoluble material in guayule rubber, which is in part fibrous and woody and hence harmful, this fraction in kok-saghyz rubber is unobjectionable. It consists principally of finely divided, soft, inert material which acts like so much filler.

Although the percentage of acetone-soluble material of kok-saghyz rubber is high, as compared with Hevea, this fraction is not resinous, or does it contribute objectionable properties such as softness or excessive tack. It consists largely of an unidentified white crystalline substance dissolved in the rubber.

In testing the physical properties of kok-saghyz rubber, both the A.C.S. and the A.C.S. alternate formulae give curing curves which are too flat for good evaluation. Satisfactory results were obtained by curing at 274° F. and adding 0.1% diphenylguanidine to the modified A.C.S. formula. The physical test data given below are therefore based on the following formula:

	T arro
Rubber	100
Zinc oxide	6
Stearic acid	
Mercaptobenzothiazole	
Diphenylguanidine	
Sulphur	3.5

A.S.T.M. recommendations were adhered to with the exception that compounding was done on a four- by eight-inch mill. Figure 2 shows a typical tensile strength-curing time curve for kok-saghyz rubber containing 0.4% of 4-4'-diaminodiphenylmethane. Some of the physical properties are as follows:

	Tested 24 Hrs. after Cure	Raw Rubber Aged Before Compounding (48 Hrs. in 0 ₂ at 70° C. & 300 P.S.I.)
Cure at 274° F.	20 min.	35 minutes
Optimum tensile	3420 p.s.i.	3100 p.s.i.
Modulus at 600%	1560 p.s.i.	1930 p.s.i.
Ultimate elongation	750%	690%
Hardness (durometer	43	44

When kok-saghyz rubber is compounded with an appropriate antioxidant, the aging properties of the vulcanized rubber compare favorably with those of Hevea. Figure 3 shows the changes in tensile strength and elongation which result when the vulcanizate from kok-saghyz rubber, compounded according to the foregoing formula plus 0.25 part of 4-4'-diphenylphenylenediamine as an antioxidant, is aged under the very severe conditions of 72 hours in oxygen at 70° C. and 300 p.s.i.

Laboratory tests on a new rubber are obviously of limited significance. Consequently the Department of Agriculture made arrangements with two large rubber companies to fabricate, in factory equipment, tires made entirely from kok-saghyz rubber produced in pilot-plant operations at the Eastern Regional Research Laboratory.

The B. F. Goodrich Co. fabricated and tested 6:00-16 fourply passenger car tires. The road tests were made with 30% overload and an operating speed of 60 miles an hour. Their report on this work states in part:

"1. Russian Dandelion Rubber is the most promising substitute for natural rubber which has thus far become available. This statement is based on a general rating of four materials as substitutes for natural rubber:

"(a) Russian Dandelion Rubber.

"(b) GR-S.

"(c) Cryptostegia Rubber.

"(d) Guayule.

"Dandelion Rubber is superior to GR-S in physical characteristics, in factory processing, in building tack, and

in most tire performance characteristics.

"Dandelion Rubber is superior to guayule in building tack. Guayule cements used as tackifying spreads on the carcass plies of GR-S tires produce unsatisfactory building characteristics. A Russian Dandelion cement used similarly produces building characteristics equal to those produced by natural rubber cements.

Russian Dandelion Rubber in tires is superior to GR-S and approaches the performance of natural rubber

in the following characteristics:

Tread wear, on the road.

"(b) Tread crack growth, on the road. "(c) General performance, on the road.

"(d) Tread crack growth, on the indoor machine. Tread tensile strength, at R.T. and 212° F.

"(f) Carcass ply adhesion.
"Russian Dandelion Rubber in tires gives performance equal to that of GR-S and of natural rubber on the Carcass Separation Test on the indoor machine."

United States Rubber Co. made and tested 9:00-20/10 rayon cord truck tires and subjected them to road tests at between 45 and 55 miles per hour with loads up to 50% above normal load. Their report states in part:

"By far the most interesting fact is that kok-saghyz carcass ran extremely well under severe conditions and we should be greatly pleased if we had a large supply of koksaghyz available now for use in the carcass.'

These comments by experts in the rubber industry leave no doubt as to the high quality of kok-saghyz rubber.

The Cost of Kok-saghyz Rubber

We have stated that kok-saghyz can be satisfactorily grown in this country, that it contains a relatively highgrade rubber, that this rubber can be isolated by a practical process, and that it can be satisfactorily fabricated into high-grade tires. The question then arises, What will kok-saghyz rubber cost? This cannot be answered at present since the largest factors in the final cost of rubber, that is, the cost of growing the roots and the yield of rubber per

acre per year, could not be adequately determined in the short time available for study. We can, however, make a reasonable estimate of the average cost of producing koksaghyz rubber from roots of an assumed rubber content delivered to the factory.

Assuming a rubber hydrocarbon content on a dry basis of 10.0% in fall-harvested roots and 13.4% in spring-harvested roots, the estimated cost of producing one pound of kok-saghyz rubber would be approximately 11.7c; a credit for the fermentable carbohydrates of 2.5c per pound of rubber would result in a net cost of 9.2c. This is based upon the harvesting cycle previously described in this article and includes amortization and all other costs except that of growing, harvesting, and transporting roots to the factory. A factory producing 10 long tons of kok-saghyz rubber per day and operating 300 days per year would serve a planting area of approximately 40,000 acres and would cost about \$1.800.000.

The foregoing assumptions are fairly conservative. Given sufficient time for breeding and selection studies, a rubber hydrocarbon content on a dry-root basis of 15% for fallharvested roots and 19.5% for spring-harvested roots might reasonably be anticipated; this would reduce the factory cost of producing a pound of rubber from 9.2 to about 7.4c. Such an increase in rubber content would likewise reduce the field costs per pound of rubber.

As already pointed out, kok-saghyz, among other rubberbearing plants, was included in the Emergency Rubber Project "to make available a source of crude rubber for emergency and defense uses." By 1944, however, it became apparent that "while the experimental data indicated that kok-saghyz holds promise of significant amounts of highgrade rubber after improvement by selection and breeding, it could not contribute sufficient quantities of rubber to emergency war requirements."4

Hence the Emergency Rubber Project work on kok-saghyz was terminated on June 30, 1944. However the Bureau of Plant Industry, Soils, and Agricultural Engineering, in its regular Rubber Plant Investigations' work, is continuing on a limited scale the selection and breeding of high rubberbearing strains of Taraxacum kok-saghyz and its cognates T. megalorrhizon (krim-saghyz), and Scorzonera tau-saghyz.

It is estimated that in time the yield of rubber per acre per year from the best of these Russian dandelion-like plants should equal or exceed that from guayule. If this be true, we might then have an annual crop which in an emergency could be expanded rapidly to yield significant quantities of high-grade natural rubber.

Wherever "natural" rubber is mentioned in this quoted excerpt, commercial grades of estate rubbers are indicated. Kok-saghyz, Cryptostegia, and guayule are all natural rubbers.
 Report of the special subcommittee of the House Committee on Agriculture, Jan. 2, 1945, House of Representatives Report No. 2098.